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(22) Application date	24 April 1991	(72) Inventor	Nakanishi
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(54) **Title of the invention:** Method for shrink-proofing of animal hair fibre products.

(57) **Abstract**

OBJECT: A durable shrink-proofing treatment is provided for animal hair fibre woven products which allows cold-water washing in a washing machine, while retaining the soft hand-feel characteristic of animal hair fibres.

CONSTITUTION: After subjecting the animal fibre woven product to low-temperature plasma treatment, a shrink-proofing treatment with shrink-proofing resin is applied, followed by a weight-reducing treatment of the animal hair fibres with a protease.

SCOPE OF CLAIM(S)

1. A method for a shrink-proofing treatment of an animal hair fibre product, characterized by a low-temperature plasma treatment of the animal hair fibre product, followed by a shrink-proof treatment with a shrink-proofing resin, and then a treatment to reduce the weight by 2-10 % with a proteolytic enzyme.

DETAILED DESCRIPTION OF THE INVENTION

Technical Field

This invention relates to a method for treating animal hair fibre products to provide durable anti-shrink properties in cold-water washing in a washing machine, without damaging the soft hand-feel of the original animal hair fibres.

Background art

When animal hair fibre products are washed in cold water, felting occurs due to the entwinement of scales which form the surface of the fibres, and there is a marked shrinkage, so generally they cannot tolerate machine washing. However, recently technology in this field has progressed and shrink-proofing treatments have been developed to tolerate machine washing, such as the Dylan method with treatment in the "top" state, the chlorinated Harcosett resin method, the Silolan-BAP method with treatment in the fabric state, and a method with a low-temperature plasma treatment, and it has become possible to obtain products that can be machine washed.

Thus, shrink properties have been somewhat improved, but after these treatments the animal hair products are rough and have a core, and the animal hairs have a hand-feel lacking in repulsion without a peculiar core.

Problem(s) to be overcome by the invention

This invention was made in view of this situation and aims at giving animal hair fibre products a high shrink resistance which can withstand 30 machine washes in cold water, without losing the soft hand-feel of the original animal hair fibres which have repulsion and draping without a core.

Means of overcoming the problem(s)

This invention achieves the above object and has the following constitution. The essence of this invention is a method for shrink-proofing of animal hair fibre product, characterized by a low-temperature plasma treatment of the animal hair fibre product, followed by a shrink-proof treatment with a shrink-proofing resin, and then a treatment to reduce the weight by 2-10 % with a proteolytic enzyme.

The invention will be described in detail in the following. In the method of this invention, a low-temperature plasma treatment is first given to the animal hair fibre product. Here, animal fibres indicates natural keratinous fibres obtained from animals such as sheep, camel, goat and rabbit, and animal hair fibre products indicates fibre products in general such as woven and knitted goods obtained with the above animal hair fibres alone or by using a mixture of the above animal hair fibres with other fibres. Examples of other fibres that can used in admixture are polyester fibres, acrylic fibres, nylon fibres, amide fibres, and polyvinyl chloride fibres.

As already mentioned, the animal hair fibre product is first treated with low-temperature plasma; the equipment to be used here for the low-temperature plasma treatment is an equipment having a construction and capacity, such that a pair of electrodes for discharge are installed in a vacuum chamber capable of maintaining a low pressure, a specified gas can be introduced at a fixed flow rate, the chamber can be evacuated by an attached vacuum pump, and a constant pressure can be maintained. When a voltage is applied to the electrodes in the vacuum chamber, a glow discharge occurs and the introduced gas comes into a plasma state. Most commonly, a high-frequency power of 13.56 MHz is applied. A low-temperature plasma treatment can be performed by passing the animal hair fibre product to be treated at a desired speed through the low-temperature plasma atmosphere formed between the electrodes.

More specifically, the product to be treated is taken into the vacuum chamber, and the internal pressure is adjusted to 0.01-10 Torr by evacuating with the vacuum pump. Then, the specified gas is introduced to adjust to 0.1-5 Torr. Various gases can be used here, such as oxygen, nitrogen, argon, helium, ammonia, air etc. When the high-frequency electric power is applied to the electrodes, the introduced gas becomes a low-temperature plasma. A suitable high-frequency power is about 0.1-5 W/cm^2 (electrode unit surface). The low-temperature plasma treatment can be performed by passing the product through this low-temperature plasma atmosphere. The time of treatment is generally from a few seconds to 300 seconds, preferably 30-180 seconds.

Next, according to the method of this invention, in order to stabilize the shrink-proofing obtained by the low-temperature treatment, a shrink-proofing resin treatment is applied to the animal hair fibre product. The shrink-proofing resin can be

block-urethan resin, polyamide epichlorohydrin resin, glyoxalic resin, ethylene-urea resin or acrylate resin. The amount of the shrink-proofing resin is preferably 0.5-10 % by weight of the fibres on solid basis. A softener can be used simultaneously with the shrink-proofing resin. When amino-modified dimethyl polysiloxan is used as softener, the hand-feel becomes soft, so this is preferred.

Application of the resin is followed by drying and heat treatment. The heat treatment is conducted at 130-160°C for about 1-2 minutes. A conventional continuous process can be used to apply the shrink-proofing resin to the animal hair fibre product.

Further, in the method of this invention, the animal hair fibre product is given a weight reducing treatment with a protein decomposing enzyme, and the hand-feel is softened. The proteolytic enzyme is a protease. Proteases may be alkaline, neutral and acidic. The protease should have the strongest possible decomposing power, and the weight loss should be as great as possible. A protease concentration of 0.5-10 % by weight is adequate. The intended softening effect can be obtained with a weight loss of about 2-10 % by weight.

The weight reducing treatment is done by the exhaustive absorption method; pH and temperature of the bath used for the treatment may be selected as the optimum conditions for the enzyme. The treatment time should be 30-60 minutes, in order to avoid surface roughness due to an extended time. Non-ionic surfactant can be used added in order to improve the penetration.

After the weight reducing treatment, a cold-water rinse and, if needed, a softener treatment with amino-modified dimethyl-polysiloxan can be conducted.

Effect

When a low-temperature plasma treatment is applied to an animal hair fibre product, a high shrink-proofing can be achieved without damaging or removing scales, but the hand-feel becomes rough. This roughening is believed to be due to an uneven surface on the scales that received the plasma action. When an animal hair fibre product in this condition is given a weight reducing treatment with protease after shrink-proofing with a shrink-proofing resin, the scales which are located in the outermost layer and are not very amenable to protection with the shrink-proofing resin, are dissolved

very easily, so the roughness disappears, the fibres become thinner, and the fibres can move more easily, so the hand-feel becomes soft. The shrink-proofing capacity combines the shrink-proofing effect of the low-temperature plasma treatment and the shrink-proofing effect of the shrink-proofing resin, and the shrink-proofing effect becomes able to withstand 30 times washing.

Working examples

In the following, the invention will be described by way of working examples; in these examples, the properties of materials were determined by the following methods.

(1) Shrinkage

JIS L-0217 (method 103, drying while hanging)

(2) Softness

JIS L-1096 (Handle-o-meter method)

(3) Hand-feel

The presence of a soft hand-feel of the animal hair having repulsion without a hard core and draping was evaluated by a perceptive test at the following 3 levels:

O : yes

Δ : slight

X : no

Working Example 1

Viyella fabric of 100 % wool 2/48 mission (?) was used, and a low-temperature treatment was conducted on this at the following low-temperature plasma condition 1.

Low-temperature plasma treatment condition 1

Type of gas	Oxygen
Gas flow rate	0.2 litre/minute
Vacuum	1.0 Torr
High-frequency output power	0.5 kW
Time of treatment	1 minute

Then, the fabric treated with low-temperature plasma was immersed in an aqueous solution according to recipe 1 below, mangled to a squeeze ratio of 70 %, and after drying at 110°C for 2 minutes, it was heat treated at 150°C for 1.5 minutes.

Recipe 1

Elastron BAP	50 g/litre
(block urethan resin, product of Dai-Ichi Kogyo Seiyaku)	
Sodium carbonate	3 g/litre
(pH adjusting agent)	
Nikka Silicone AM-3000	20 g/litre
(product of Nikka Kagaku, amino-modified dimethyl polysiloxan)	

Next, weight reduction was conducted by enzyme treatment with a liquid according to Recipe 2 below, a bath ratio of 1:30, 50°C for 30 minutes in a liquid-flow dyeing machine. The weight reduction ratio was 4 %.

Recipe 2

Entilon PN-10L	15 % owf
(protease, product of Rakuto Kasei)	
Actinol R-100	1 g/litre
(product of Matsumoto Yushi, nonionic surfactant)	

It was then rinsed in cold water, rinsed in hot water, treated with Nikka Silicone AM-3000 (product of Nikka Kagaku, amino-modified dimethyl polysiloxan) at a bath ratio of 1:30, 40°C for 10 minutes, and dried after water removal.

For the purpose of comparison, a comparative fabric was obtained by the same method as in Working Example 1, except that the enzyme treatment in this example was omitted.

Properties of the shrink-proofed fabrics of the invention and the comparison were measured and evaluated, and the results are shown in Table 1.

Table 1

		Invention	Comparison
Shrinkage after 30 times washing (%)	Warp	1.3	1.2
	Woof	1.1	1.0
Softness (g)	After finishing	190.2	221.5
	After 30 times	175.3	200.6
Hand-feel		○	X

As seen from Table 1, the fabric shrink-proofed according to this invention had a softer hand-feel and had durable shrink-proof properties, while maintaining the hand-feel of the animal hair fibre product.

Effect of the invention

According to the method of this invention, good shrink-proof properties which withstand 30 cold-water washes can be conferred on animal hair fibre products, while maintaining a soft hand-feel having repulsion without a hard core and draping.